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CHEMICAL AND MINERALOGICAL ANALYSIS FOR PROVENANING OF THE BRONZE AGE POTTERY FROM SHAHR-I-SOKHTA, SOUTH EASTERN IRAN

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ABSTRACT

The present study has typologically selected fifteen potsherds from Bronze age site of Shahr-i-Sokhta (Iran) analysed by X-Ray Fluorescence (XRF) and Scanning Electron Microscopy (SEM) with Energy Dispersive X-Ray Spectroscopy (EDS) to determine the mineralogical, chemical, and morphological characteristics of the pottery samples. Thin-Section Petrography is also utilized to evaluate the XRD and XRF results and to identify the texture and the geographical status of the samples and X-Ray Diffraction (XRD) is used to determine their mineralogy. The clay fabric was fine and common texture is of high siliceous matter and include calcareous and non-calcareous clay types with high refractory. The presence of diopside, albite and analcime minerals in the shards points to a firing temperature that lies in the 900°C in an oxidizing atmosphere. It is concluded that all Shahr-i-Sokhta pottery shard samples could be described as local pottery with sandy clay from the same source.

KEYWORDS: Shahr-i-Sokhta pottery, XRF, Thin-Section Petrography, SEM (EDS), XRD, XRF, clays, prehistory

1. INTRODUCTION

As a prehistoric site belonging to the Bronze Age, Shahr-i-Sokhta—located at southeast of Iran in a state called Sistan, see Fig. 1—is comprised of four periods and ten cultural phases that makes its cultural remains worthy of research and analysis (Table I). Earlier excavations of the area, and the subsequent huge number of discovered pottery fragments, reveal that pottery had a fundamental role in the lives of its residents. Preliminary studies on these fragments also indicate that the potteries are wheel-made (Vidale & Tosi, 1996) and in the majority of the cases, the color of the paste is buff. Buff ware is indeed the dominant pottery at Shahr-i-Sokhta, ranging from an absolute buff to green. From the statistical point of view gray ware is the second in diffusion and red ware is the third (Tosi, 1983; Sarhadi, 2013). Based on the shapes, Shahr-i-Sokhta's pottery can be divided into four large groups: bowls, jars, beakers and dishes (Rahman, 2013).



Figure 1. Map of Shahr-i-Sokhta, Sistan area from Iran (Rahman, 2013).

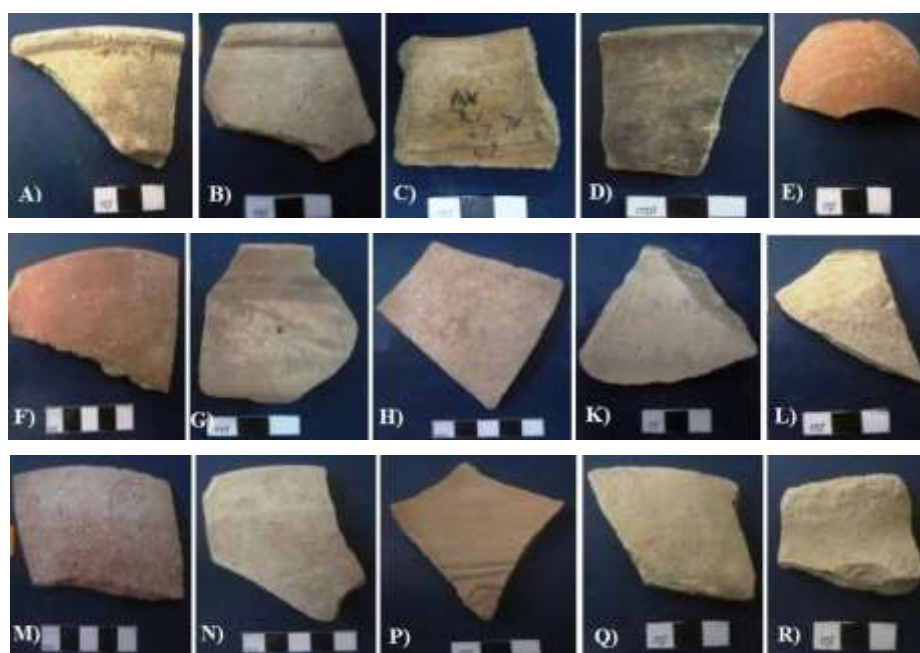


Figure 2. Pottery shard samples from Shahr-i-Sokhta.

This study investigates the compositional analyses of a set of pottery from Shahr-i-Sokhta that are found in the first archaeological investigation by Sir Arul Stein. In addition, the Italian archaeological expedition of IsMEO (International Association of Mediterranean and Oriental Studies), headed by Piperno and Tosi (1975), Tosi (1983) and Salvatori and Vidale (1997) started extensive excavation and typological research on its pottery assemblage randomly between 1967 and 1978 (Piperno & Tosi, 1975; Amiet, Tosi, & Meriggi, 1978). The second cycle of systematic excavation and archaeological research, such as the excavation of kilns and workshops on this site

was done by Sajjadi in 2003 (Sajjadi et al., 2003). Clearly, the existing studies on these fragments mainly focus on typological analysis and though by identifying their shape, decoration and manufacturing techniques, they have expanded the boundaries of archaeological knowledge, they have left the compositional examination of these potteries unattended. The present essay is then a timely attempt to carry out a compositional survey on the pottery of Shahr-i-Sokhta by aiming to: a) identify the compositional features of these potteries; b) find out whether the chemical and morphological analyses could be utilized as the pottery of Shahr-i-Sokhta with regard

to their manufacturing and firing temperature; and finally, c) by linking the identified raw materials of the potteries to the geographical characteristics of the area, determine whether the potteries are local or imported. To achieve these objectives, this study will focus on the chemical and mineralogical analyses of the potsherds. To this end, fifteen pottery shards, which are similar in terms of style and chronology, are taken as samples to be examined, see Figure 2.

Table I. Chronological chart of Shahr-i-Sokhta (Piperno & Tosi, 1975).

Period	Phase	Radiocarbon dates	Cultural
IV	0	2000-1800 B.C.	Late Bronze Age
	1		
	2		
III	3	2500-2300 B.C.	Middle Bronze Age
	4		
II	5	2700-2600 B.C.	Early Bronze Age
	6		
	7		
I	8	3300-3200 B.C.	Late Chalcolithic
	9		
	10		

The samples are collected randomly from the Zahedan Museum in Iran, where there were not any advanced laboratories available to analyze the samples. Subsequently, the compositional analyses of this limited number of potsherds are carried out in laboratory at the Center of Global Archaeological Research in University Science Malaysia, Penang. The findings of this study not only will disclose the compositional features of the potteries, they will also pave the ground for further comparative archaeological studies of Shahr-i-Sokhta area with similar neighboring sites that belong to the Bronze Age.

3. RESULT

3.1 X-RAY FLUORESCENCE (XRF) ANALYSIS

X-ray fluorescence (XRF) is employed to examine the chemical data of the fifteen pottery shard samples. Each powder sample weighing 0.4 g is mixed until homogenous with the flux powder of a type of Spectroflux 110 (product of Johnson and Matthey) and is heated for one hour at 105°C temperature. These mixtures are baked for one hour in a furnace with a temperature of 1100°C. Homogenous molten is molded into fused glass with a thickness of 2 mm and a diameter of 32 mm. Molten pallet is analysed using a fully computerized AXios mAX Model PANALYTICAL X-ray fluorescence at the Laboratory of the Centre for Global Archaeological Research (CGAR), University of Science Malaysia (USM), Penang. The major constituents of the samples are: SiO₂, Al₂O₃, TiO₂, Fe₂O₃, MgO, MnO, CaO, Na₂O, and K₂O (Table II). The chemical composition determines the type of clay as calcareous or non-calcareous and as well as low or high refractory in nature. The calcareous or non-calcareous type is identified by the percentage of calcium oxide (CaO) in each pottery shard. Based on the chemical composition, if clay contains >6% CaO, it is taken as calcareous clay and with CaO < 6% as non-calcareous type. To survey the refractory, fluxes (K₂O, FeO, CaO, MgO and TiO₂) is < 9%, the clays are classified as local clay with high refractory in nature (Maniatis and Tite, 1981). Based on the x-ray fluorescence, Shahr-i-Sokhta pottery shard samples are calcareous clay, yet sample 6(C) with a 1.51% CaO and sample 7(E) with only 5.38% of CaO are known as non-calcareous type. In addition, all shahr-i-Sokhta pottery shard samples are flux (K₂O, FeO, CaO, MgO and TiO₂) >9% which points their being low refractory in nature. However, the fluxes help melt silicates and bind the particle of clay together during firing.

Table II. X-Ray fluorescence analysis of pottery shard samples from Shahr-i-Sokhta.

Sample No	Element %								
	SiO ₂	Al ₂ O ₃	CaO	Fe ₂ O ₃	MgO	MnO	TiO ₂	Na ₂ O	K ₂ O
1	53.94	11.67	8.50	4.55	6.32	0.10	0.49	3.77	1.54
2	52.63	12.29	11.97	4.75	5.90	0.10	0.52	2.62	2.05
3	58.07	11.72	9.24	4.47	6.08	0.10	0.48	2.51	2.14
4	51.45	10.80	11.06	4.33	5.65	0.10	0.47	2.51	2.41
5	52.66	12.48	10.76	4.65	5.96	0.10	0.50	2.48	1.59
6	61.45	15.31	1.51	6.41	3.63	0.09	0.75	2.00	2.81
7	56.40	15.66	5.38	6.38	3.85	0.10	0.71	1.77	2.74
8	52.42	11.61	9.46	4.55	4.07	0.10	0.51	2.22	2.91
9	54.83	12.26	13.47	4.68	3.27	0.05	0.67	1.20	2.41
10	55.51	11.49	9.87	4.47	6.25	0.09	0.50	3.41	2.50
11	55.92	11.92	11.32	4.73	6.95	0.09	0.50	2.49	2.52

12	57.38	16.80	6.71	7.18	4.16	0.09	0.78	1.67	3.37
13	51.95	12.60	13.28	5.05	6.63	0.09	0.54	2.24	2.62
14	54.99	11.48	9.59	4.58	6.60	0.09	0.47	2.83	2.84
15	57.27	16.22	6.08	6.77	4.18	0.10	0.73	2.22	3.36

3.2 THIN-SECTION PETROGRAPHY ANALYSIS

Nine samples are created by sawing off a small fragment of the material. The resulting samples are grounded and then attached to the flat surface of a microscope glass slide to expose the surface of the fragment to a standard thickness (25-30 micrometers). At this stage, the identification of minerals under the polarized-light microscope is based on optical and morphological properties that is carried out using a BA 300 Motic and MX 9430 Meiji petrographic (polarizing) microscopes at the Centre for Global Archaeological Research (CGAR), University of Science Malaysia (USM), Penang. The resulting samples are grounded and then attached to the flat surface of a microscope glass slide to expose the surface of the fragment to a standard thickness (25-30 micrometers).

At this stage, the identification of minerals under the polarized-light microscope is based on optical and morphological properties. The observations of the samples revealed that the major presence of angular and sub angular quartz (SiO_2), that is also explained by the results of the X-ray diffraction analysis. The presence of fine to medium size of quartz grains in angular shapes is believed to be formed due to the grinding process on the mixture of clay and sand. Other identified minor minerals include feldspars (albite), pyroxene (diopside and augite) and opaque iron oxide that are each present to an approximate amount of 10%. Drawing on Wentworth's size chart, it is easy to deduce that the combination of iron oxide and grains of pyroxene is also mixed with fine-sand, silt fraction, and grains (up to 0.07 mm and 0.1 mm respectively), see Figure 3.

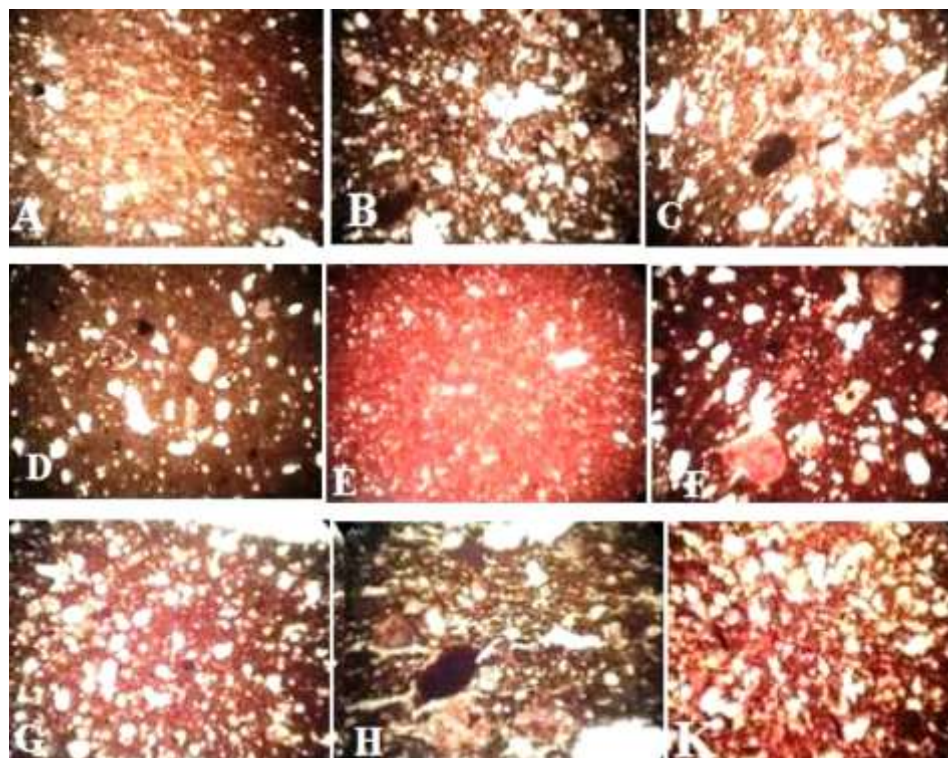


Figure 3. Thin-section Petrography (Scale 1mm; observed in optical crystallography and petrographic microscope); (Magnification 40X): A) fine and medium grained quartz and feldspar mineral fragments, quartz + feldspar rock fragments; B) fine and medium grained quartz and feldspar and pyroxene mineral fragments, quartz + feldspar rock fragments; C) Quartz mineral and feldspar (plagioclase); D) fine and medium grained quartz and feldspar and pyroxene mineral fragments, quartz + feldspar rock fragments; E) abundant very fine grained quartz minerals, red iron-oxide stained clay clots, and a few coarser-grained quartz mineral fragments; F) feldspar rock fragments (plagioclase) and pyroxene mineral in crystalline groundmass of quartz and iron oxide; G) fine and medium grained quartz and feldspar and iron-oxide mineral fragments; H) feldspar rock fragments (plagioclase) and pyroxene mineral in crystalline groundmass of quartz and iron oxide; K) fine and medium grained, sub angular and angular quartz feldspar, and pyroxene fragments and dark red iron-oxide.

3.3 SCANNING ELECTRON MICROSCOPY ANALYSIS

To characterise the fifteen pottery shard samples from Shahr-i-Sokhta, SEM analysis is carried out using FEI QUANTAFEG 650 Model equipped with EDS (Energy Dispersive a X-ray Spectrometer) by OXFORD Model from England at the laboratory of the Centre for Global Archaeological Research (CGAR), University of Science Malaysia (USM), Penang. In SEM analysis, electron beam is focused on a small area of the pottery shard surface, secondary x rays are emitted at wavelengths characteristic of the particular element that is excited. The scanning electron microscopy provides information on the type of the used clay in the making of the potteries. This would help us understand the variation in the percentage of the composition of the minerals of the pottery shard samples. This method is beneficent in that it made the study of the samples possible without destroying them (Table III). In addition, SEM observation confirms the results found through the mineralogical (XRD) and chemical (XRF) analyses. The SEM-EDS spectrum of each sample exhibits the presence of elements like Silicon, Aluminum, Potassium, Iron, Magnesium and Sodium as the predominant constituents of the shards, though the percentage of the composition of the minerals varies. Microphotograph and EDS spectra of present shape

Samples are shown in Fig 4 and 5. SEM analysis confirmed the XRF results that all pottery shard are calcareous (>6%) except samples C and E which are non-calcareous (<6%) type.

The EDS spectrum of each sample is determined that Shahr-i-Sokhta pottery shards are contained high percentage of silica oxide (SiO_2), and lesser amount of aluminum oxide (Al_2O_3), iron oxide (Fe_2O_3), calcium oxide (CaO), potassium oxide (K_2O) and magnesium oxide (MgO) that is identified the organic matter present in these pottery shards. On the other hand, same elements found in the Shahr-i-Sokhta pottery shard samples is the particular evidence that potters used locally available clay to manufacture pottery and the technology of production was a sophisticated one. In general, pottery shard samples thus seem to be produced from illite/Kaolin whose chemical composition contains SiO_2 , Al_2O_3 , MgO , CaO and Fe_2O_3 with low refractory in nature that are fired in oxidizing atmosphere in the range of 850-950°C. The microphotograph of Shahr-i-Sokhta shards discloses fine texture of silica-rich components such as sand (quartz, feldspar and rock fragments), with smooth, homogeneous, and small crystalline sizes that are observed in the high melting temperature. Furthermore, the smooth surface observed in the samples is due to the reflective nature caused by early sintering.

Table III. SEM results of pottery shard samples from Shahr-i-Sokhta.

Sample No	Element %							
	SiO_2	Al_2O_3	CaO	FeO	MgO	TiO_2	NaO	K_2O
1	40.90	13.95	14.88	8.56	6.52	1.05	4.71	2.49
2	8.22	2.46	2.66	0.75	1.28	0.90	0.72	0.40
3	64.18	15.08	14.86	12.34	8.81	0.66	2.45	7.17
4	53.56	14.67	11.82	5.74	7.46	0.63	2.72	5.28
5	48.84	14.13	15.64	6.53	7.79	0.57	2.67	1.87
6	62.13	16.27	2.19	7.78	4.71	0.76	2.18	3.58
7	52.61	12.54	4.80	16.78	3.33	3.97	1.45	5.80
8	68.74	12.30	18.88	5.95	7.09	0.78	2.25	3.96
9	48.41	13.57	11.85	4.11	8.19	1.39	2.65	6.96
10	50.55	12.66	26.12	5.36	7.86	0.44	3.14	2.75
11	45.06	9.59	15.44	14.21	10.76	-	3.51	1.52
12	62.47	18.36	6.95	7.87	3.38	1.00	2.10	4.44
13	47.44	13.52	21.46	18.39	9.70	1.09	1.67	3.54
14	63.29	17.02	12.81	4.71	6.79	-	1.91	2.45
15	62.66	16.69	7.89	0.90	8.09	3.34	2.67	8.47

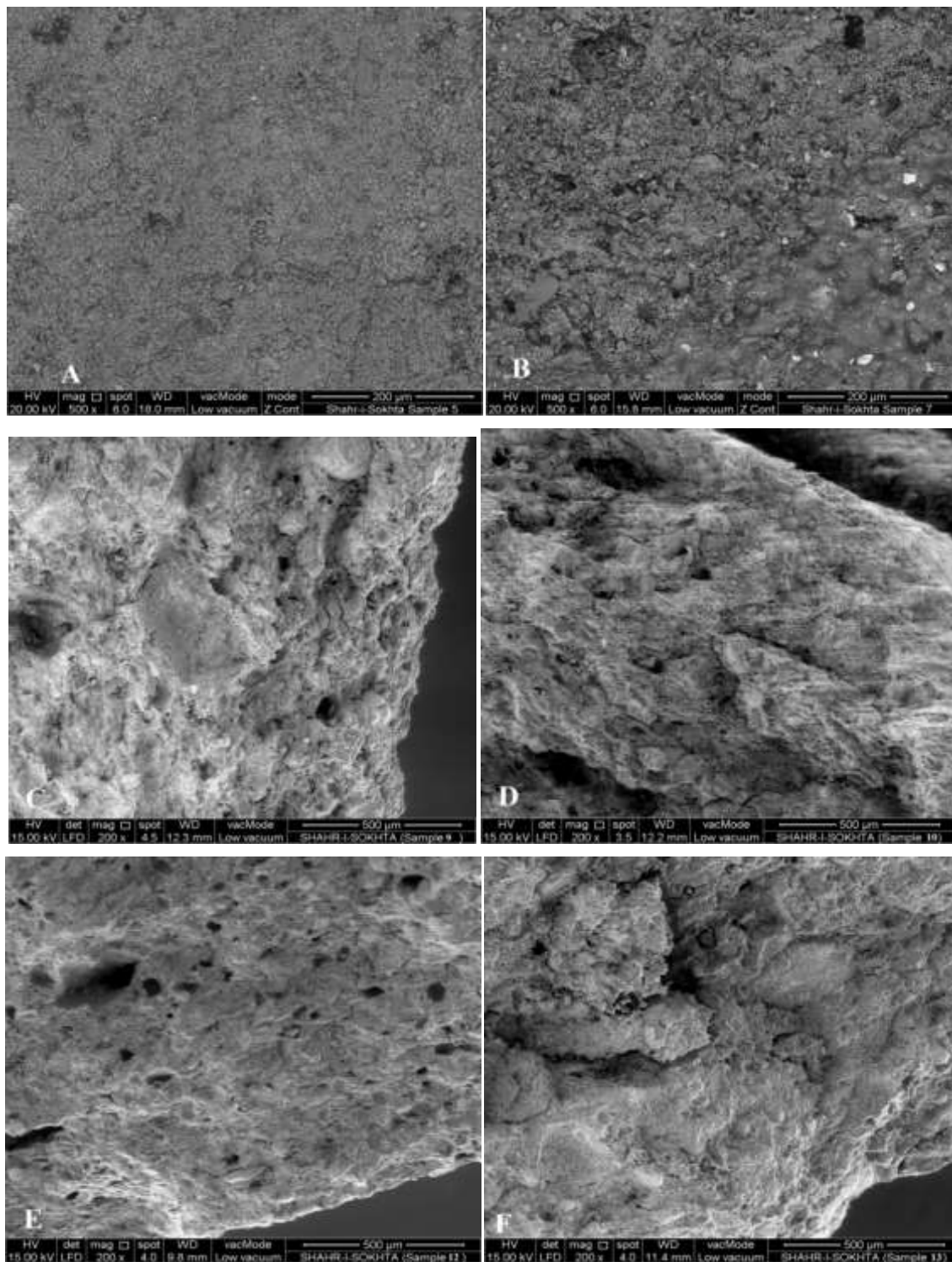


Fig 4 Micro photographs of Shahr-i-Sokhta pottery shard samples (500mm): A) Unglazed, Fine and smooth ware, quartz grain, Alumina-silicate (feldspars), (image taken at 20 kv, 18.0 mm working distance and 500 magnification); B) Unglazed, Fine and smooth ware, Quartz grain, Alumina-silicate (feldspars), (image taken at 20 kv, 15.8 mm working distance and 200 magnification); C) Unglazed, Large and medium quartz grain, Alumina-silicates (feldspars), Alkali feldspar, Ca carbonate calcium, High iron oxide and magnesium (image taken at 15 kv, 12.3 mm working distance and 200 magnification); D) Unglazed, Medium and small quartz grain, Alumina-silicates (feldspars), Alkali feldspar, Ca carbonate calcium (image taken at 15 kv, 12.2 mm working distance and 200 magnification); E) Unglazed, Fine and smooth ware, quartz grain, Alumina-silicate (feldspars), High iron oxide and magnesium (image taken at 15 kv, 9.8 mm working distance and 200 magnification); F) Unglazed, Large and medium quartz grain, Alumina-silicates (feldspars), Alkali feldspar, Ca carbonate calcium (image taken at 15 kv, 11.4 mm working distance and 200 magnification).

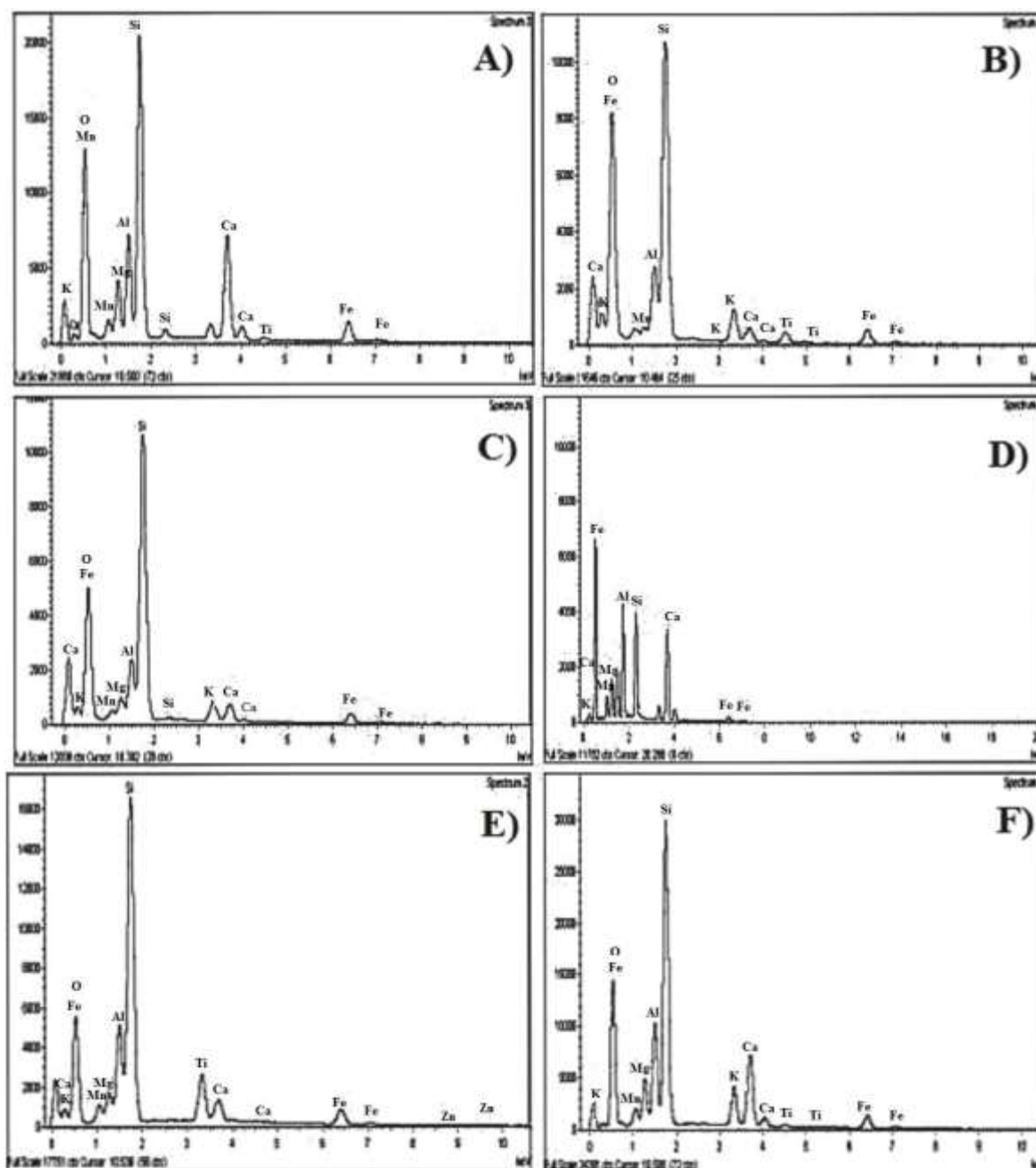


Figure 5. EDS spectrum of Shahr-i-Sokhta pottery shard samples; A) High composition contains SiO_2 , Al_2O_3 , MgO , CaO and Fe_2O_3 (calcareous type); B) High composition contains SiO_2 , Al_2O_3 , MgO , and Fe_2O_3 and low percentage of CaO (non-calcareous type); C) High composition contains SiO_2 , Al_2O_3 , MgO , and Fe_2O_3 and low percentage of CaO (non-calcareous type); D) High composition contains SiO_2 , Al_2O_3 , MgO , CaO and Fe_2O_3 (calcareous type); E) High composition contains SiO_2 , Al_2O_3 , MgO , and Fe_2O_3 and low percentage of CaO (non-calcareous type); F) High composition contains SiO_2 , Al_2O_3 , MgO , CaO and Fe_2O_3 (calcareous type).

3.4 X-RAY DIFFRACTION (XRD) ANALYSIS

To carry out the X-Ray Diffraction (XRD) analysis, 20 mg of powdered pottery is pressed into a glass slide and is inserted in the X-ray diffraction machine D8 ADVANCE Model BRUKR Company from Germany at the CGAR, USM, Penang, to determine the mineralogical composition. The semi-quantitative mineralogical results of the same samples are shown in Table IV. The main identified minerals are quartz,

feldspars (albite), analcime, and pyroxenic (diopside and augite). The presence or absence of any of these specific mineral assemblages is often employed to estimate the firing temperature of pottery (Mirti & Davit, 2001; Papachristodoulou et al., 2006). Nevertheless, the presence of quartz is not surprising at all as this phase has been found in many ancient pottery shards. The presence of diopside can be related to a rather high firing temperature, since this calcium

silicate starts to crystallize in calcareous clays by the analysis of calcium carbonate at a temperature of nearly 900°C. On the other hand, the presence of feldspar (albite) in most of the samples indicates that all samples were fired at around 900°C.

In addition, analcime shown in the Shahr-i-Sokhta pottery shards is often combined from chemical reagents such as aluminum silicate at high temperature. The presence of augite pyroxene and absence of calcite in the Shahr-i-Sokhta potsherds might indicate that the primary calcite was completely decomposed

upon firing at a temperature of at least 900°C. Based on the XRD results, the higher amount of iron oxide (hematite and muscovite) in the samples 6 and 7 as non calcareous types, together with the absence of pyroxene or feldspar, have caused the pyroxene or feldspar not able to affect iron oxide to bleach the color of pottery shards after firing; and hence, these samples are known as red ware. In general, all pottery shards (calcareous and non- calcareous) are fired in the range of 850-900°C.

Table IV. X-Ray diffraction of pottery shard samples from Shahr-i-Sokhta.

Sample No	Mineral (%)						
	Quartz	Albite	Diopside	Analcime	Augite	Hematite	Muscovite
1	6.22	14.13	70.52	4.58	-		
2	5.58	10.91	81.12	2.39	-		
3	23.25	10.56	54.66	5.28	25.60		
4	4.24	15.72	79.88	0.16	-		
5	1.43	11.28	78.02	9.27	-		
6	77.27	16.93	-	-		5.80	
7	6.24	-	61.38	3.07	-		2.83
8	56.37	12.12	13.98	-	-		
9	85.45	-	14.55	-	-		
10	28.49	24.53	-	11.09	40.55		
11	11.64	7.48	56.68	-	12.59		
12	6.39	-	-	6.11	-		
13	28.96	23.64	40.33	-	-		
14	34.38	20.39	20.72	3.28	14.46		
15	33.12	19.42	31.29	-	-	-	-

4. DISCUSION

Based in the chemical results, Shahr-i-Sokhta pottery shard samples are divided in to calcareous and non- calcareous (samples 6 and 7 Figures C and E) clay and also the fluxes concentration higher than 9% therefore, Shahr-i-Sokhta pottery shards are low refractory in nature with the same source. According to the petrography analysis, Shahr-i-Sokhta shards are comprised of the quartz grains with the angular and sub angular shape point out that the sand was crushed before being added to the clay. Crushed quartz is typically used to strengthen the ware. The rounded grains, in turn, were either natural inclusion in the clay or were added without crushing or sieving. The results of thin-section analysis of Shahr-i-Sokhta pottery show that all the samples contain natural components such as quartz mineral as well as feldspar, pyroxene and opaque iron oxide. The minerals and rock fragments utilized in the Shahr-i-Sokhta pottery are very typical of the geological environment of the Sistan area, suggesting that the pottery was locally made.

In addition, Shahr-i-Sokhta pottery shards are distinguished on the basis of kaoline/illite components with variety quartz, potash feldspar, iron ox-

ide, sandy clay, and sediment, which are derived from local place. Furthermore, the samples are included of quartz, pyroxene, feldspars and specially analcime that are suggested the shards are fired between 850- 900°C in oxidizing. It is already discussed that the high percentage of iron oxide, as hematite, in sample 6, and the muscovite in sample 7 with less CaO of Shahr-i-Sokhta samples is a forming feature of red ware.

It should be noted, pottery composition depends both in the clay source and in the recipe used to prepare the clay paste. Thus, the abundance ratios of some elements may be altered as a result of mixing of several materials. Scanning Electron Microscopy (SEM), used to evaluate the results of the other two methods (XRF and XRD), indicates that high percent of Alumina (Al_2O_3) existing in potshards has led to the formulation of many attractive properties like a high degree of hardness and strength, good and effective thermal pottery. On the other hand, EDS spectrum and microphotograph reveal that Shahr-i-Sokhta samples are comprised of quartz, feldspars, sand and rock sediment in the small crystalline size that are smooth and homogenous in high temperature.

5. CONCLUSION

In conclusion, Sistan area is a large desert with some huge mountains, lakes, rivers and a delta area. The Helmand Basin is one of the largest rivers that flow into this region. The area presents a limited variation in terms of local contributions of sediments of Helmand River to the geological formation of the Shahr-i-Sokhta area. In addition, the variation of lakes sediments, and grain and sand from rocks is reflected on the different compositional raw materials of the Shahr-i-Sokhta pottery. With respect to the local contributions of sediments, the potteries are hence richer in CaO contents due to the proximity of the Shahr-i-Sokhta rock limestone. Other pebbles or rocks, which are coated by a dark and shiny varnish, consist of iron oxide (Fe_2O_3) which is one of the minerals in the Shahr-i-Sokhta pottery samples and is spread over the stones from the basaltic block mountain by the wind. As a refractory material content, the amount of the zirconium is also higher than other minerals, which might be linked to the igneous phases, coming from the granite and pegmatite regional rocks, the sedimentary ones that are abundant throughout the area. The quite uniform chemical profile implies that potteries were probably produced locally.

On the other hand, the increased magnesium and Ca content is probably due to the certain cations (Mg, Na, and K) in water from the lakes and the Helmand River found in the Shahr-i-Sokhta site (Tosi, 1983). In general, these Deposit and fine grain (silt and clay) materials along the terminal lakes, having been dried out of the Helmand delta basin, are carried away by Aeolian erosion.

In spite of this, more research on the weathering and geology effects is still needed in that these may be called sandy clay that are typically used as rock salt structure. In this context, relatively high concentrations of Na is attributed to the geochemical affinity of these elements with the rock salt of Helmand River in the surrounding of Shahr-i-Sokhta in the Sistan area (Tosi, 1983).

And finally, the existing amount of aluminum in the samples point to their high degree of hardness, strength and good thermal. All the same, it is to be noted that despite the inevitable limitation of this study—having access to a limited number of samples and having to transport them and carry out the analysis in a foreign country—the acquired data of Shahr-i-Sokhta pottery is quite beneficial for future studies on the site, particularly an investigation over the effect of geological position on the pottery in the prehistoric periods or a comparative study of Shahr-i-Sokhta with neighboring or similar sites.

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